



**ELEMENTAL ANALYSIS
OF
TURBINE ENGINE GAS PATH CLEANERS**

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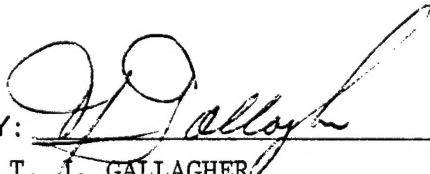
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INTRODUCTION

The military specification, MIL-C-85704 requires that the gas path cleaners (cleaning compounds) used for turbine engine compressors, should be screened for high levels of corrosive elements. The concentration of these corrosive elements such as sodium, potassium, sulfur, phosphorus and chloride is very critical, and should be maintained below the specified values. The high concentrations of these contaminants, if left behind, can cause corrosion of engine components and/or degradation of coatings and ceramic materials.

Three analytical methods have been selected for the required element analysis of cleaning compounds. Atomic absorption spectroscopy (AA)¹ is a well known and a very specific technique with few interferences. These interferences and the means to deal with them are also well defined.^{2,3} AA is very sensitive for sodium and potassium determinations in organic matrix.⁴⁻⁶

Inductively coupled plasma atomic emission spectroscopy (ICP-AES) has made considerable progress during the last ten years and has become a recognized analytical tool for multi-element analysis. A large number of references and reviews are available in the literature.⁷⁻¹⁴ The routine wet chemical, spectrophotometric and other analytical methods for sulfur¹⁵⁻¹⁸ and phosphorus^{19,20} are tedious and time consuming. The ICP-AES method has been used successfully for the determination of sulfur and phosphorus in low concentrations.²¹⁻²³

Chloride is a most common corrosive contaminant and several methods are available for its determination in various products.²⁴⁻³² Electrochemical methods such as polarography, amperometry, potentiometry (ion selective electrode) etc. are the most sensitive methods, but are limited in their use to aqueous solution analysis only. A microcoulometric titration (MCT) method³³ has been studied in detail for the chloride content of cleaning compounds.

Sample preparation techniques such as chemical separation, matrix modification or acid digestion could introduce undesirable contamination or loss of analyte species. This sample preparation step also increases the time of analysis, therefore, no sample pretreatment should be required to perform an efficient analysis. The three selected analytical methods provided required accuracy, sensitivity and precision necessary for monitoring elemental analysis of gas path cleaners with minimal sample preparation.

Per MIL-SPEC the maximum allowed values for elements present in the gas path cleaners are,

Sodium	50 mg/L
Potassium	50 mg/L
Phosphorus	50 mg/L
Sulfur	500 mg/L
Chlorine	100 mg/L
Other trace elements	10 mg/L

This report will present the complete elemental analysis results for gas path cleaners.

EXPERIMENTAL PROCEDURES

Instruments:

Atomic Absorption Spectrophotometer, Perkin-Elmer 3030

Single element Hollow Cathode Lamps (HCL) for Co, Cu, Fe, K, Mg, Mn, Na, Pb, Si and V.

Inductively Coupled Plasma Atomic Emission Spectrophotometer, Perkin-Elmer ICP/6000.

Micro Coulometric Titration System, Dohrmann-Xertex MCTS-20

REAGENTS

All reagents used were AnalR grade. Standard solutions for calibration were prepared by serial dilution of Spex AA grade stock solutions. Deionized distilled water was used for the preparation of all standard and sample solutions. The gas path cleaners studied were proprietary products and will be referred to as samples A, B and C. Samples were diluted with deionized distilled water for AA and ICP-AES analysis. Chlorobenzene in iso-octane or toluene was used as the chloride standard solution. Pharmco grade ethyl alcohol was used as sample diluent in microcoulometry.

Acid digestion with nitric and sulfuric acids mixture, and Parr bomb digestion methods were used to prepare aqueous solutions of the cleaning compounds. These solutions were analyzed for the comparison of data obtained from direct sample analysis.

All standard and sample solutions were stored in polyethylene bottles for the duration of analysis period.

OPERATING PARAMETERS

The analytical wavelengths used for the determination of sodium, potassium, sulfur, phosphorus and other trace contaminants using AA and ICP-AES methods are reported in Table 1. Detection limits of AA and ICP-AES methods for these elements is also presented in Table 1. Instrumental set-up and experimental parameters for AA analysis are presented in Tables 2 and 3 respectively. Experimental conditions used for ICP-AES analysis are reported in Table 4. Parameters for the chloride determination are presented in Table 5.

RESULTS

Data obtained from atomic absorption analysis of gas path cleaners for sodium and potassium is presented in Tables 6 and 7. Samples were diluted 50, 60, 80, 100, 125, 250 and 500 times for sodium, and 2.5, 5.0, 7.5, 10, 50, 75, 100, 125 and 250 times for potassium determinations with deionized distilled water. Each reported value is an average of 5 to 10 readings.

The standard addition method, which is an extremely valuable tool in the AA analysis of complex matrix samples, was also used. In this method, a sample was spiked with a known amount of standard analyte solution. If some material was present in the sample causing either a matrix, chemical or ionization interference, it would have reduced the absorbance of analyte to be determined in the unspiked sample. However, the absorbance increase from the added analyte in the spiked

sample would also be reduced by the same proportional amount, since the concentration of the interferent remained the same in the unspiked and spiked sample solutions. Results from direct analysis and standard addition methods were comparable to within $\pm 2.0\%$ error, as can be seen from the data reported in Tables 6 and 7.

Acid digested and Parr bomb digested samples were analyzed for sodium and potassium as well as for other trace elements such as cobalt, copper, iron, magnesium, manganese, lead, silicon, vanadium, etc. Results for these elements are recorded in Table 8.

The performance of the ICP-AES technique was tested for the elemental analysis of gas path cleaners. Comparative data for direct and acid digested sample analyses by AA and ICP-AES methods is presented in Table 9. The values for all the elements determined by direct analysis of the samples (diluted with deionized distilled water to make 10% solution) were found to be slightly lower than the acid digested samples by both AA and ICP-AES methods. Figure 1 shows the standard calibration curves for all the elements. Corrections for acid blank were applied to all the values. Results obtained by the two methods are complimentary to each other. It was found that the ICP-AES method was not very sensitive for sodium and potassium determinations.

For the determination of sulfur and phosphorus, most of the available AA methods are indirect procedures, whereas, ICP-AES is a direct method and it requires less than an hour for complete analysis. Results for sulfur, phosphorus and silicon determinations by ICP-AES are reported in Table 10.

The results for the chloride determinations by the micro coulometric titration method are provided in Table 11. Direct analysis of samples as well as analysis after sample dilution with ethanol gave similar results. The data obtained from this method was compared with other methods. Samples prepared by Parr bomb digestions were analyzed by direct titration using micro-coulometry, ion selective electrode and ion chromatography methods. Results are presented in Table 12.

CONCLUSIONS

Elemental analysis of gas path cleaners by AA, ICP-AES and MCT methods prove that the concentration of sodium, potassium, sulfur, phosphorus chloride and other trace elements can be determined routinely. These methods are simple and efficient.

RECOMMENDATIONS

It is recommended that AA, ICP-AES and MCT methods be used for the required gas path cleaner analysis: Atomic absorption for sodium and potassium, inductively coupled plasma atomic emission for sulfur, phosphorus, vanadium, lead and silicon, Micro-coulometric titration for chloride and AA and ICP-AES for trace elements.

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TABLE 1
SELECTED WAVELENGTHS FOR AA AND ICP-AES ANALYSIS

ELEMENTS	ANALYTICAL WAVELENGTH, nm		DETECTION LIMIT, mg/L	
	AA	ICP-AES	AA	ICP-AES*
Cobalt	240.7	238.89	0.01	0.006
Copper	324.75	324.75	0.002	0.007
Iron	248.3	238.20	0.004	0.005
Lead	217.0	220.35	0.03	0.042
Magnesium	285.2	279.07	0.0003	0.001
Manganese	279.5	257.61	0.05	0.002
Phosphorus	213.6	213.68	20.0	0.05
Potassium	766.5	404.72	0.002	42.85
Silicon	251.60	251.60	0.06	0.08
Sodium	589.0	558.95	0.0005	0.029
Sulfur	—	180.73	—	?
Vanadium	318.4	309.31	0.04	0.005

*Detection limits in accordance with International Union of Pure & Applied Chemistry (IUPAC) recommendations

TABLE 2

INSTRUMENTAL SET-UP PARAMETERS FOR AAS

ELEMENTS	WAVELENGTH nm	SLIT nm	FLAME	SENSITIVITY mg/L	OPTIMUM RANGE mg/L
Cobalt	240.7	0.2	Air/C ₂ H ₂	0.01	3.5
Copper	324.75	0.7	Air/C ₂ H ₂	0.05	5.0
Iron	248.3	0.2	Air/C ₂ H ₂	0.04–0.1	5.0
Lead	217.0	0.7	Air/C ₂ H ₂	0.03	20.0
Lead (flow spoiler)	283.3	0.7	N ₂ O/C ₂ H ₂	—	2.7
Magnesium	285.2	0.7	Air/C ₂ H ₂	0.005	0.5
Manganese	279.5	0.2	Air/C ₂ H ₂	0.04	3.0
Potassium	766.5	0.7	Air/C ₂ H ₂	0.02	2.0
Phosphorus	213.6	0.2	N ₂ O/C ₂ H ₂	250–290	10,000.0
Silicon	251.6	0.2	N ₂ O/C ₂ H ₂	0.8–2.0	150.0
Sodium	589.0	0.2	Air/C ₂ H ₂	0.001	1.0
Vanadium	318.4	0.7	N ₂ O/C ₂ H ₂	0.6–2	100.0

TABLE 3

PARAMETERS FOR AAS ANALYSIS

ELEMENT	LAMP CURRENT mA	BURNER HEIGHT mm	FUEL/ OXIDANT RATIO	CHARACTER- ISTIC CON- CENTRATION (CHAR. CONC.)	CALCU- LATED CHAR. CONC.	ABSOR- BANCE
Cobalt	30	8.8	18/55	0.1200	0.1179	0.229
Copper	20	8.0	21/44	0.0770	0.0874	0.248
Iron	18	9.5	18/45	0.1000	0.1070	0.203
Lead	10	8.6	20/40	0.1900	0.1944	0.444
Magnesium	20	9.3	20/46	0.0078	0.0080	0.276
Manganese	15	8.0	24/47	0.0520	0.0444	0.199
Potassium	15	8.0	20/40	0.0430	0.0440	0.219
Sodium	12	9.5	20/47	0.0120	0.0130	0.33
Vanadium	15	7.2	32/38	1.9000	1.8958	0.236

TABLE 4

EXPERIMENTAL CONDITIONS FOR ICP-AES ANALYSIS

Plasma	Argon, high purity gas
Plasma flow rate	12 ml/min.
Auxillary flow rate	1 l/min.
Plasma height	15 mm
Nebulizer	22-25 psi
RF Generator, 2500 W	operated at 27.12 MHz
Incident Power	1250 for aqueous solutions 1500 for organic solutions
Reflectance Power	< 10 w
Purge gas for S & P	Nitrogen

TABLE 5

INSTRUMENTAL PARAMETERS FOR MICRO-COULOMETRY

FURNACE TEMPERATURE SETTINGS:

Inlet	700°C
Center	800°C
Outlet	800°C

GAS SETTINGS:

Reactant gas (O)	160 ml/min.
Carrier gas (He or Ar)	40 ml/min.

COULOMETER SETTINGS:

Bias	250 mV
Gain	1100-1200
Time	300 secs. (adjustable)

TABLE 6

AAS ANALYSIS OF GAS PATH CLEANERS FOR SODIUM

DILUTION FACTOR	SAMPLE A		SAMPLE B	SAMPLE C
	SODIUM IN mg/L		SODIUM IN mg/L	SODIUM IN mg/L
	DIRECT ANALYSIS AFTER DILUTION	STANDARD ADDITION METHOD		
50x	—	—	7.65	37.5
60x	—	—	7.56	35.3
80x	—	—	8.7	33.2
100x	101.7	98.9	7.9	35.6
125x	104	105.7	8.7	41.8
250x	108	109.5	7.5	42.5
500x	115	109.3	7.0	33.7
Average	107.2	105.9	7.14	37.09

TABLE 7

AAS ANALYSIS OF GAS PATH CLEANERS FOR POTASSIUM

DILUTION FACTOR	SAMPLE A		SAMPLE B	SAMPLE C
	POTASSIUM mg/L		POTASSIUM IN mg/L	POTASSIUM IN mg/L
	DIRECT ANALYSIS	STANDARD ADDITION METHOD		
2.5x	3.06	2.6	—	—
5.0x	2.5	2.8	—	—
7.5x	3.1	3.3	—	—
10.0x	3.3	3.5	—	—
50.0x	—	—	121.80	2.85
75.0x	—	—	123.50	2.68
100.0x	—	—	128.60	2.39
125.0x	—	—	118.80	4.25
250.0x	—	—	132.50	3.08
Average	3.06	3.05	124.88	3.08

TABLE 8

AAS ANALYSIS OF ACID DIGESTED GAS PATH CLEANERS

NAME OF ELEMENTS AND SAMPLES	CONCENTRATION mg/L	STANDARD DEVIATION	COEFFICIENT OF VARIATION (%)
Cobalt			
Sample A1	1.79	0.013	2.62
Sample A2	1.73	0.022	2.22
Sample B	1.30	0.012	2.84
Sample C	2.52	0.011	2.00
Copper			
Sample A1	0.88	0.002	2.80
Sample A2	0.99	0.003	2.30
Sample B	0.58	0.003	3.79
Sample C	2.07	0.003	1.07
Iron			
Sample A1	0.55	0.017	1.32
Sample A2	0.60	0.018	1.99
Sample B	5.00	0.015	1.66
Sample C	20.16	0.021	0.94
Magnesium			
Sample A1	1.46	0.001	0.49
Sample A2	1.02	0.004	1.20
Sample B	1.27	0.001	0.32
Sample C	1.52	0.002	0.64
Manganese			
Sample A1	0.07	0.009	7.67
Sample A2	0.10	0.031	2.11
Sample B	0.099	0.005	6.09
Sample C	0.20	0.004	3.66
Potassium			
Sample A1	3.57	0.005	0.85
Sample A2	3.64	0.008	3.77
Sample B	124.00	0.010	0.70
Sample C	3.29	0.008	1.00
Sodium			
Sample A1	102.45	0.001	1.87
Sample A2	108.06	0.002	1.47
Sample B	9.45	0.002	4.72
Sample C	34.4	0.001	1.63

TABLE 9

COMPARATIVE DATA FROM AAS AND ICP-AES METHODS

ELEMENTS AND SAMPLES	AA METHOD		ICP-AES METHOD	
	NO SAMPLE PREP mg/L	ACID DIGESTED SAMPLE mg/L	NO SAMPLE PREP mg/L	ACID DIGESTED SAMPLE mg/L
Calcium				
Sample A1	3.0	3.25	3.3	3.33
Sample A2	2.85	3.0	3.0	3.15
Sample B	0.9	0.87	0.6	0.62
Sample C	1.0	1.06	0.71	0.80
Cobalt				
Sample A1	1.71	1.79	1.65	1.70
Sample A2	1.68	1.73	1.56	1.58
Sample B	1.30	1.30	1.2	1.23
Sample C	2.50	2.52	2.39	2.50
Copper				
Sample A1	0.75	0.88	0.55	0.60
Sample A2	0.89	0.99	0.75	0.78
Sample B	0.47	0.58	0.52	0.59
Sample C	2.0	2.07	2.48	2.6
Iron				
Sample A1	0.38	0.55	0.30	0.34
Sample A2	0.60	0.6	0.67	0.72
Sample B	4.95	5.0	5.48	5.85
Sample C	21.02	20.16	15.46	16.48
Magnesium				
Sample A1	1.41	1.46	1.51	1.55
Sample A2	1.0	1.02	1.12	1.17
Sample B	1.22	1.27	0.63	0.7
Sample C	1.48	1.57	0.99	1.12
Manganese				
Sample A1	0.06	0.07	0.10	0.07
Sample A2	0.08	0.1	0.08	0.06
Sample B	0.10	0.099	0.04	0.06
Sample C	0.13	0.2	0.10	0.12

TABLE 10

SILICON, SULFUR AND PHOSPHORUS BY ICP-AES

SAMPLES	SILICON, mg/L		SULFUR, mg/L		PHOSPHORUS, mg/L	
	DIRECT METHOD	STANDARD ADDITION	DIRECT METHOD	STANDARD ADDITION	DIRECT METHOD	STANDARD ADDITION
Sample A	2.5	2.05	ND*	ND*	5.24	6.66
Sample B	0.4	0.42	120.0	119.86	8.0	8.72
Sample C	0.8	0.75	130.0	130.09	9.0	8.88

*ND — not determined

TABLE 11

CHLORIDE IN GAS PATH CLEANERS BY MICRO-COULOMETRIC TITRATION

SAMPLE NAME	CHLORIDE mg/L	STANDARD DEVIATION
Sample A, as it is	61.1	2.57
Sample A, 2x dilution with ethanol	63.0	0.42
Sample A, 10x dilution with ethanol	61.0	0.52
Sample B, as it is	26.3	0.11
Sample B, 2x dilution with ethanol	25.0	0.33
Sample B, 10x dilution with ethanol	24.0	0.57
Sample C as it is	16.6	1.19
Sample C, 2x dilution with ethanol	16.3	0.76
Sample C, 10x dilution with ethanol	17.0	0.48

TABLE 12

COMPARATIVE DATA FOR CHLORIDE IN GAS PATH CLEANERS

ANALYTICAL METHOD USED	SAMPLE A, mg/L	SAMPLE B, mg/L	SAMPLE C, mg/L
Direct furnace injection micro-coulometric titration. No sample preparation.	61.7	25.1	16.63
Direct micro-coulometric titration after Parr Bomb digestion.	63.0	25.5	25.0
Ion Selective Electrode method after Parr Bomb digestion.	65.0	24.0	14.9
Ion Chromatography after Parr Bomb digestion.	60.0	26.0	20.7
Furnace injection micro-coulometric titration method after Parr Bomb digestion and ion exchange treatment.	62.6	27.3	17.5

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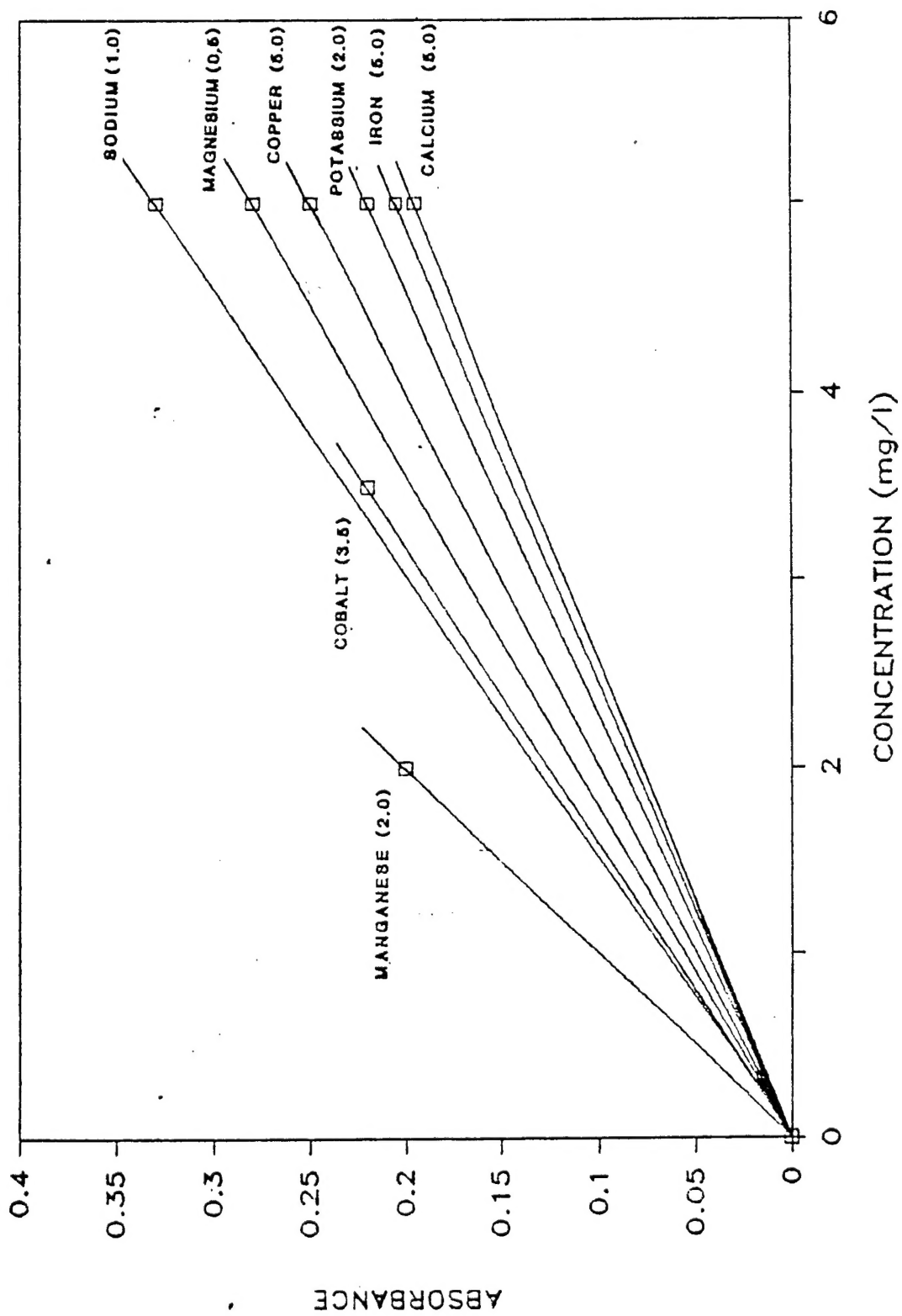


Figure 1. Calibration Curves for AA Analysis

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